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proportions of the 3 different types of N from an expanded region XPS for the N1s peak. It was determined that the amount of active N sites of the total N sites was only 40%. This then allowed the amount of lithium sulphite and, similarly, the amount of transition metal oxide sink to be calculated accurately, thereby avoiding redundant components.

## Cell Test Data

Cells were tested in two modes:

1. Asymmetric and;
2. Symmetric.

## 4C—Test of Cell in Asymmetric Mode

In this test mode, a TR-PAN and  $\text{Li}_2\text{SO}_3$  based cathode was combined with an anode containing TR-PAN and CuO as a lithium sink. Following the formation step, cells were cycled at constant currents varying from 1 mA of 60 mA and the DC capacitance determined from the product of the inverse of the slope of the voltage-time (V-t) plots. FIG. 6 shows the cycling behaviour of the TR-PAN/ $\text{Li}_2\text{SO}_3$ /CuO asymmetric supercapacitor; it shows typical saw tooth behaviour for a lab cell charged and discharged at a constant current of 1 mA. The cell delivered a reversible unit capacity of over  $0.16 \text{ mAh cm}^{-2}$  and capacitance of  $0.32 \text{ F cm}^{-2}$  to 2.5 V. (For a pure EDLC supercapacitor, typical values of  $0.05 \text{ mAh cm}^{-2}$  and  $0.1 \text{ F cm}^{-2}$  would be expected).

FIG. 7 shows the rate performance of the asymmetric supercapacitor, namely, the variation of electrode unit capacitance with current density. After an initial sharp drop in unit capacitance, there is a slow decrease from  $0.32 \text{ F cm}^{-2}$  to  $0.164 \text{ F cm}^{-2}$  for a 40 times increase in current density. This indicates very good energy retention at high power densities for the electrodes.

FIG. 8 illustrates the cycle life performance of the asymmetric supercapacitor. Over 10,000 cycles were recorded without any loss of reversibility.

## 4D—Test of Cell in Symmetric Mode

In this test mode following formation, cells are disassembled and the previously formed cathodes were used as both electrodes in test cells. FIG. 9 shows the Rate performance of the symmetric supercapacitor i.e. the variation of electrode capacitance with current density for the symmetric supercapacitor. The capacitance at similar current densities is higher than was observed for the asymmetric cell and the decrease in capacitance with increasing currents is less. This indicates better electrode matching and power performance in the symmetric than asymmetric cells.

Although in Example 4, CuO was used as the sink, other transition metal oxides such as SnO, NiO could also be used as a  $\text{Li}^+$  sink. SnO is a suitable alternative sink compound and gives rise to an electrode with adequate performance. NiO which has similar physical properties to CuO and SnO, such as reduction potential and weight, would be equally suitable. Electrodes formed using an intercalation compound  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  as a sink produced less capacitance as electrodes, probably because more of the electrode is taken up with the parasitic weight of the titanate structure, whereas the metal oxides at least contribute to conductivity when they are left as a metallic residue. Cyclic voltammogram experiments recorded at a scan rate of 10 mV/s showed electrodes with CuO, SnO and  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  sinks in the anode delivered a specific capacitance of 400 F/g, 350 F/g and 370 F/g, respectively at 3.5 V (vs  $\text{Li}^+/\text{Li}$ ).

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## Example 5

## Control Example—Preparation of Anode and Cathode for High Energy Supercapacitors (without a Sink in Anode)

## 5A—Preparation of Anode Components

TABLE 3

Anode components	
Material	Composition by weight (% w/w)
TR-PAN (active material)	69.5
Activated carbon (AC)	14.5
Denka Black (carbon additive)	6
PVDF (binder)	10

TR-PAN, activated carbon and Denka Black powders were dried separately at  $120^\circ \text{C}$ . under vacuum for 2 hours. 24 g of TR-PAN, 5 g of activated carbon and 2 g of Denka Black were then ground and mixed (or bead milled using a planetary mixer) and added to 28.6 g of a 12 w/w % solution of PVDF in NMP with constant stirring using a T50 overhead mixer for 1-2 hours until a smooth and homogenous viscous composite ink (or slurry) was obtained. 70 g of excess NMP was added to achieve the desired viscosity and consistency. The ink was cast onto Al foil to form a coating using a Doctor-Blade technique. In the first instance, the wet coatings were dried at  $100^\circ \text{C}$ . for 2 hours to remove excess solvent, followed by  $120^\circ \text{C}$ . under dynamic vacuum to expel remaining traces of solvent. After evaporation of the solvent thin composite films were obtained.

Electrodes of a geometrical area of  $1 \text{ cm}^2$  were punched out of the coated sheets and assembled into lab cells with a  $\text{Li}_2\text{SO}_3$  based cathode. A porous glass fibre (Whatman), Celgard 3501 or PTFE based separator was used to electronically separate the cathode from the anode, and either 1M  $\text{LiBF}_4$  (lithium tetrafluoroborate) and tetraethyl ammonium tetrafluoroborate ( $\text{NEt}_4\text{BF}_4$ ) in propylene carbonate (PC) or 1.5M  $\text{LiBF}_4$  in  $\gamma$ -butyrolactone/ethylene carbonate (GB/EC) mixture was used as the electrolyte. Ink preparation and cell assembly were carried out in a dry room.

## 5B—Preparation of Cathode Components

TABLE 4

Cathode components	
Material	Composition by weight (% w/w)
TR-PAN (active material)	56.5
Activated carbon (AC)	14.1
Denka Black (carbon additive)	6
$\text{Li}_2\text{SO}_3$ ( $\text{Li}^+$ source)	13.4
PVDF (binder)	10

Lithium sulphite ( $\text{Li}_2\text{SO}_3$ , synthesised according to the first aspect), TR-PAN, activated carbon and Denka Black powders were dried separately at  $120^\circ \text{C}$ . under vacuum for 2 hours. 13.4 g of  $\text{Li}_2\text{SO}_3$ , 20 g of TR-PAN, 5 g of activated carbon and 2 g of Denka Black were then ground and mixed (or bead milled using a planetary mixer) and added to 29.5 g of a 12 w/w % solution of PVDF in NMP with constant stirring using a T50 overhead mixer for 1-2 hours until a smooth and homogenous viscous composite ink (or slurry) was obtained. 65 g of excess NMP was added to achieve the desired viscosity and consistency. The ink was cast onto Al foil to form a coating using a Doctor-Blade technique. In the first instance,